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I, KIM MARSHALL, MANAGER EXAMINATION SUPPORT AND SALES,
hereby certify that the annexed is a true copy of the Provisional specification as filed
on 7 April 1997 in connection with Application No. PO 6050 for a patent by JAMES
COOK UNIVERSITY OF NORTH QUEENSLAND.

I further certify that the annexed specification is not, as yet, open to public inspection.



WITNESS my hand this Fifteenth
day of April 1998

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AUSTRALIAN	
PROVISIONAL No.	DATE OF FILING
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PATENT OFFICE	

JAMES COOK UNIVERSITY OF NORTH QUEENSLAND
&
CSR LIMITED

AUSTRALIA
Patents Act 1990

PROVISIONAL SPECIFICATION

Invention Title: FOOD GRADE WAX AND PROCESS FOR
 PREPARING SAME

This invention is described in the following statement:

This invention relates to a wax composition suitable for use in comestibles. The invention also relates to a process for preparing the composition.

Many comestibles include waxes added during preparation of the comestible. For example, wax is a component of chewing gum base. Waxes can also be used as protective coatings on comestibles such as cheeses and fruits. The waxes used for such purposes are typically mineral waxes such as montan wax extracted from lignites, peat waxes, ceresin wax and petroleum waxes. Among these mineral waxes, it appears that only petroleum based waxes are used in food applications: microcrystalline waxes, high melting point waxes and high sulfur microcrystalline waxes in particular. The US Food and Drug Administration (FDA) has established regulations for the use of petroleum wax (see 21 CFR 172.886 and 178.3710) and Japanese authorities consider petroleum waxes to be natural products and allows their use in products such as chewing gum. Although montan wax is not used directly in food applications, it is used in plastic processing such as plastic containers and wrappings which will come or may come into contact with food.

Use of mineral waxes in comestibles is undesirable. Mineral waxes are extracted from coal and crude petroleum oil. These raw materials contain organic chemicals which are toxic to humans. The food applicability of the waxes depends on the degree of refining or purification achieved and its usage has been regulated according to the specifications provided by authorities such as Ministry of Agriculture, Fisheries and Food, UK (The Mineral Hydrocarbons in Food Regulation, SI 1966 No. 1073. This regulation applies to England and Wales only, though similar regulations apply to Scotland and Northern Ireland). The refining achieved has been acceptable to food regulating authorities. However, recent studies have shown toxicological effects of petroleum based waxes ("Recommendations on the use of mineral hydrocarbon in food", Food Advisory Committee 8/93, UK) and the

hydrocarbon imparted by packaging using mineral hydrocarbon waxes on food (Progress Report of the Working Party on Chemical Contaminants from Food Contact Materials: 1988 to 1992; Food Surveillance Paper No. 38, Ministry of Agriculture of Fisheries and Food). The Ministry of Agriculture, Fisheries and Food is at present reviewing studies on the toxicological impact of using petroleum based waxes as food additives and as a processing aid to update regulation on the usage of these waxes in food applications (Ministry of Agriculture of Fisheries and Food News Release 278/93 of 19 August 1993).

Waxes of plant origin are known. Indeed, the palm *Copernicia cerifera* is a source of the common wax, carnauba. Waxes can also be extracted from sugar cane and rice. Subject to the conditions used for extraction, waxes of plant origin should provide an alternative to mineral waxes for use in comestibles.

Sugar production results in a number of waste products, one of which is mill mud. Mill mud comprises crude wax and fats, fibre, sugar, crude protein and ash (SiO_2 , CaO , P_2O_5 and MgO). A crude cane wax can be extracted from mill mud. However, the crude wax is unsuitable for use in comestibles as it has a foul odour and taste and is dark green to brown in colour due to the presence of contaminants. US Patent No. 2,464,189 describes a process for the refining of sugar cane wax. However, wax produced by this process is unsuitable for use in comestibles for the following reasons:

1) The refining process is not complete. The process is only a fractionation step which removes a resinous fraction (pitch) from the sugar cane wax. The wax colour still has to be removed and stabilised. The patentees suggested further processing - for example, bleaching with acid, decolourisation, emulsification (see Example 3, line 40).

2) The reagent used in the process (acetone) is not food grade.

3) Bleaching uses reagents such as chromic and sulfuric acid which are not food grade reagents.

Since waste products of sugar production are plentiful in countries such as Australia, it would be desirable to have a process for producing a wax from such waste (ie. mill mud) suitable for use in comestibles.

The object of this invention is thus to provide a sugar cane wax composition, and a process for preparing the composition, which wax is suitable for use in comestibles.

In one aspect, the invention provides an essentially odourless and colourless wax composition obtainable from sugar cane, the composition comprising on a weight basis: wax esters, 7.2 - 7.7%; alkyl ketones, 2.6 - 2.8%; tri-glycerides, 0.5 - 0.6 %; alcohol, 1.8 - 10.2%; and, free fatty acids and polar lipids, 79.4 - 87.2%.

In a second aspect, the invention provides a process for preparing a wax composition from crude sugar cane wax, the process comprising the steps of:

- i) heating a solution of the crude wax with a lower alcohol as solvent at the boiling point of the solvent;
- ii) allowing phase separation of the solution from (i) and decanting the upper phase while hot;
- iii) allowing the separated phase from (ii) to cool and separating precipitated wax from the solvent;
- iv) repeating steps (i) to (iii) using the wax from (iii) until all pitch has been removed from the wax; and
- v) heating the wax to between 90 and 120°C and oxidising molten wax by sparging with air, oxygen or ozone.

As indicated above in the description of the second aspect, the method of refining the crude sugar cane wax involves heating the crude wax with an organic solvent to allow the pitch and the paler wax fraction to separate. These form two distinct phases, which can be separated by decantation. The paler wax fraction is cooled to allow the wax to crystallise and separate from the oil which

remains soluble in the organic solvent. The wax is filtered until dry. The wax is melted over a hot bath and oxidised by blowing fine air bubbles through a sparger. This is continued until oxidation is complete noticeable by lack of any further colour change.

The solvent used in step (i) of the process is typically ethanol or iso-propanol which have boiling points of 78.5°C and 82.4°C, respectively. The crude wax is typically combined with solvent at a ratio of one part (by weight) wax to nine parts solvent although ratios of 1:8 to 1:20 can also be used. Heating can be for 5 to 60 minutes although a heating time of 30 minutes is usually adequate.

As indicated above, the upper phase formed in step (ii) contains the wax of interest. The lower phase is a resinous fraction referred to as "pitch" which contains wax of lower quality.

In step (iii), filtration or centrifugation are advantageously used to separate wax from oil-containing solvent. However, other methods known to those of skill in the art can be used.

Steps (i) to (iii) are repeated from 2 to typically no more than 5 times. The number of times the steps are repeated largely depends on the amount of pitch present in the crude wax, the rate with which the pitch settles and the rate of wax crystallisation. It appears that pitch that does not settle fast enough is occluded within the wax crystals.

In the final step of the process, oxidation can be enhanced by using a catalyst. Suitable catalysts include cobalt or manganese borates and resinates (A.J.C. Andersen, *Refining of Oils and Fats for Edible Purposes*, Second Revised Edition, P.N. Williams, ed., Pergamon Press, 1962).

The crude sugar cane wax can be prepared by methods known to those of skill in the art. A suitable method is described, for example, in US Patent No. 2,508,002, the entire content of which is incorporated herein by cross

reference. A brief description of a suitable process follows.

Crude Wax Extraction

Sugar filter cake is steam heated and charged to a continuous reactor where it is mixed with solvent (naphtha) and held at the desired temperature and pressure. Wax is extracted into the solvent, which is subsequently separated from the filter cake. The separated wax containing solvent is then passed through a flash drum and an evaporator to separate the crude wax from the solvent. The resulting filter cake is then steam stripped to recover residual solvent.

The composition of crude sugar cane wax is typically as presented in Table I.

Table I
Composition of Crude Sugar Cane Wax

Component	Composition (% w/w)
Wax Ester	5.9-8.5
Alkyl Ketone	3.2-1.6
Tri-Glyceride	0-1.6
Alcohol	7.9-8.3
Free Fatty Acid/Sterol	5.9-7.8
Polar Lipid	73.2-76.1

Advantages of the wax composition according to the invention and the process for preparing the composition are as follows:

1) The process produces a potential food grade vegetable wax product, which could be used as a replacement for mineral waxes in a number of food or non-food applications, including (but not limited to) chewing gum base, cheese coating, fruit coating.

2) The process is simple and of low cost and

enables economic use of wax for applications indicated in (1).

3) The wax product is colourless or has low colour (pale yellow) and little or no odour and taste.

5 4) The wax product has a hardness comparable to carnauba wax.

5) The wax product has good temperature stability as compared to other vegetable waxes, such as carnauba and rice wax.

10 Having broadly described the invention, examples of the preparation of wax composition will now be given.

Example 1

One hundred grams of crude sugar cane wax was
15 combined with 900 grams of ethanol in a round bottom flask. The mixture was heated in a heating mantle to 78.5 °C for 30 minutes under reflux. The solution was removed from the heating mantle and the phases of the pitch and the paler wax are allowed to separate. The paler wax was
20 decanted into another round bottom flask while the wax mixture was still in solution or only partially crystallised at 65 to 75 °C. The mixture was again heated to 78.5 °C for 10 minutes. The paler wax was decanted to separate it from the pitch. These heating and separation
25 processes are repeated about four times or until no visible pitch separates from the paler wax. The pitch was reheated with about 100 grams of ethanol to recover additional paler wax. This is combined with the previously collected paler wax. The paler wax was then
30 cooled slowly to room temperature and then in an ice bath. The cooled wax was filtered and remelted in a hot bath between the temperature of 90 to 120 °C. Air was blown into the molten wax by means of a sparger until oxidation was complete or until there was no further
35 visible change in the colour of the wax. The overall process can be executed within several hours depending on the temperature and air distribution.

Wax composition produced by the above process had

the following properties: pale yellow in colour with a sweet smell and little or no taste.

Example 2

One hundred grams of crude sugar cane wax was combined with 900 gram of isopropanol in a round bottom flask. The mixture was heated in a heating mantle to 82.4°C. The mixture was cooled slowly to room temperature and then in an ice bath. The wax was filtered and transferred into another round bottom flask. To the wax, 450 grams of ethanol was added and the mixture heated to 78.5°C for 30 minutes. The fractionation and oxidation that follows was as in Example 1.

Wax composition produced by the above process had the following properties: pale yellow in colour with a sweet smell and little or no taste.

Yields of fractions produced using processes such as described in Examples 1 and 2 are presented in Table II.

Table II
Fractionation Yield

20	Fraction	Yield
	Oil	25%
	Pitch	35%
	Wax Composition	40%

It will be appreciated that many changes can be made to the processes and compositions as exemplified above without departing from the broad ambit and scope of the invention.

DATED THIS 7TH DAY OF APRIL 1997

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By their Patent Attorneys

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